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IS 1448-107 (1982): Methods of test for petroleum and its products, Part 107: Precipitation number of lubricating oils using the cold-cranking simulator [PCD 1: Methods of Measurement and Test for Petroleum, Petroleum Products and Lubricants]

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“Knowledge is such a treasure which cannot be stolen”

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IS : 1448 [P : 107] - 1982
(Reaffirmed 2006)

Indian Standard
METHODS OF TEST FOR PETROLEUM AND ITS PRODUCTS
[P : 107]
PRECIPITATION NUMBER OF LUBRICATING OILS

(First Reprint DECEMBER 1998)
UDC 621.892.096 : 543.862.3

Adapted with permission from ASTM Designation D 91-80.

1. SCOPE

1.1 This method covers the determination of the precipitation number of steam cylinder stocks and black oils, and may be used for other lubricating oils.

2. SIGNIFICANCE

2.1 Fully refined petroleum oils normally contain no naphtha insoluble material. Semi-refined or black oils frequently contain some naphtha insoluble material (sometimes referred to as asphalts). This test measures the amount of naphtha insoluble material in the oil. This quantity is reported as the precipitation number.

3. TERMINOLOGY

3.1 Precipitation Number of Lubricating Oils — The number of millilitres of precipitate formed when 10 ml of lubricating oil is mixed with 90 ml of precipitation naphtha, and centrifuged under prescribed conditions.

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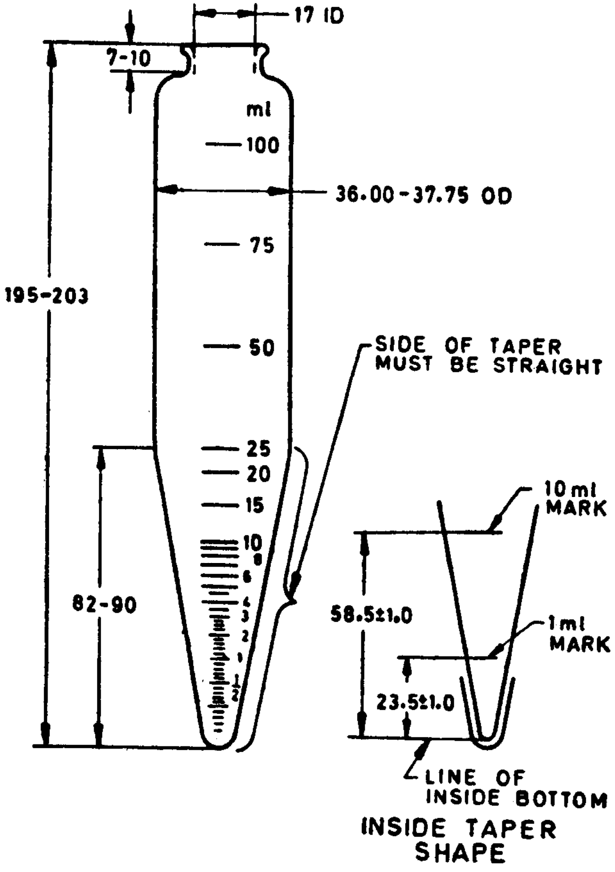
4. APPARATUS

4.1 Centrifuge Tube — Cone-shaped, conforming to the dimensions given in Fig. 1, made of glass and thoroughly annealed. The graduations, numbered as shown in Fig. 1, shall be clear and distinct, and the mouth shall be constructed in a shape suitable for closure with a cork. Scale-error tolerance and smallest graduations between various calibration marks are given in Table 1 and apply to calibrations made with air-free water at 20°C.



All dimensions in millimetres.

FIG. 1 CONE-SHAPED CENTRIFUGE TUBE

TABLE 1 CALIBRATION TOLERANCES FOR 200 mm CENTRIFUGE TUBE

(*Clause* 4.1)

Range (1) ml	Subdivision (2) ml	Volume Tolerance (3) ml
0 to 0·1	0·05	± 0·02
Above 0·1 to 0·3	0.05	± 0·03
,, 0·3 ,, 0·5	0·05	± 0·05
,, 0·5 ,, 1·0	0·10	± 0·05
,, 1·0 ,, 2·0	0·10	± 0·10
,, 2·0 ,, 3·0	0·20	± 0·10
,, 3·0 ,, 5·0	0·5	± 0·20
,, 5·0 ,, 10	1·0	± 0·50
,, 10 ,, 25	5·0	± 1·00
,, 25 ,, 100	25	± 1·00

4.2 Centrifuge — Meeting all the safety requirements for normal use and capable of whirling two or more filled centrifuge tubes at a speed which can be controlled to give a relative centrifugal force (rcf) between 600 and 700 at the tip of the tubes. The revolving head, trunnion rings, and trunnion cups, including the rubber cushion, shall be soundly constructed to withstand the maximum centrifugal force capable of being delivered by the power source. The trunnion cups and cushions shall firmly support the tubes when the centrifuge is in motion. The centrifuge shall be enclosed by a metal shield or case strong enough to eliminate danger if any breakage occurs. Calculate the speed of the rotating head by means of the following equations:

$$\text{rpm} = 1337 \sqrt{rcf/d}$$

where

rcf = relative centrifugal force, and

d = diameter of swing, in mm, measured between tips of opposite tubes when in rotating position.

4.2.1 Table 2 shows the relationship between diameter swing, rcf, and revolutions per minute.

TABLE 2 ROTATION SPEEDS FOR CENTRIFUGES OF VARIOUS DIAMETERS

(*Clause* 4.2.1)

DIAMETER OF SWING* (1)	rev/min at 600 rcf (2)	rev/min at 700 rcf (3)
mm		
48	1 490	1 610
51	1 450	1 570
53	1 420	1 530
56	1 390	1 500

*Measured in millimetres between tips of opposite tubes when in rotating position.

5. REAGENT

5.1 Precipitation Naphtha — Shall conform to the following requirements (*see* Note).

Test	*Requirement*	*Method of Test, Ref to IS : 1448**
Density at 15°C	0·692 to 0·702	P : 16
Aniline point	58 to 60°C	P : 3
Initial boiling point	Not less than 50°C	P : 18
50 percent point	70 to 80°C	P : 18
End point	Not more than 130°C	P : 18

NOTE — *Caution* — For safety precautions, *see* Appendix A.

6. PROCEDURE

6.1 Measure a 10-ml sample of the oil to be tested in each of two clean, dry centrifuge tubes at room temperature. Fill each tube to the 100-lm mark with the naphtha (*see* Note) and close tightly with softened cork (not a rubber stopper). Then invert each tube at least 20 times, allowing the liquid to drain thoroughly from the tapered tip of the tube each time. Place the tubes in a water bath at 32 to 35°C for 5 minutes. Momentarily remove the corks to relieve any pressure, and invert each tube again at least 20 times exactly as before. The success of this method depends to a large degree upon having a thoroughly homogeneous mixture which will drain quickly and completely from the tapered tip when the tube is inverted.

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*Methods of test for petroleum and its products.

NOTE — *Caution* — Naphtha is an extremly flammable liquid. For safety precautions *see* Appendix A.

6.2 Balance the two certifuge tubes or pairs of tubes with their respective trunnion cups and place them on opposite sides of the centrifuge head. Then whirl them for 10 minutes at a rate sufficient to produce a relative centrifugal force (rcf) between 600 and 700 at the tips of the whirling tubes (*see* **4.2**). Repeat this operation untill the volume of sediment in each tube remains constant for three consecutive readings. In general, not more than four whirlings will be required for oils having a low precipitation number.

7. CALCULATION AND REPORT

7.1 Read the volume of the solid sediment at the bottom of each centrifuge tube, estimating to 0·1 ml or closer if possible. If the two readings differ by not more than 0·1 ml, report the mean of the two as the 'Precipitation Number'. If the two readings differ by more than 0·1 ml, make two more determinations and report the average of the four determinations.

8. PRECISION

8.1 Results of duplicate tests shall not differ by more than the following amounts:

Precipitation Number	*Repeatability*	*Reproducibility*
0·00 to 1·20	10 percent of the mean	30 percent of the mean

APPENDIX A

(*Clauses* 5.1 *and* 6.1)

A-1. PRECAUTIONARY STATEMENT — PRECIPITATION NAPHTHA

Danger

Extremely flammable. Harmful if inhaled vapours may cause flash fire. Keep away from heat, sparks and open flame. Keep container closed.

Use with adequate ventilation. Avoid build up of vapours and eliminate all sources of ignition especially nonexplosion proof electrical apparatus and heaters. Avoid prolonged breathing of vapour or spray mist. Avoid prolonged or repeated skin contact.

Reprography Unit, BIS, New Delhi, India